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By authority of *CSAR* Date *3-31-74*
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RESEARCH MEMORANDUM

THERMAL STABILITY OF DECABORANE AND OF A
COMMERCIAL ETHYL DECABORANE (HEF-3) IN
THE RANGE 202° TO 252° C

By G. E. McDonald

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**NATIONAL ADVISORY COMMITTEE
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WASHINGTON

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RESEARCH MEMORANDUM**CLASSIFICATION CHANGED**

THERMAL STABILITY OF DECABORANE AND OF A COMMERCIAL ETHYL

DECABORANE (HEF-3) IN THE RANGE 202° TO 252° **UNCLASSIFIED**

By G. E. McDonald

By authority of *ESTAR* Date *3-31-71*

SUMMARY

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The thermal stabilities of a commercial grade of ethyl decaborane (HEF-3) and of decaborane were investigated at temperatures of 202° and 252° C. The products of decomposition are hydrogen and a boron hydride, which is a solid or viscous liquid. Ethyl decaborane also yields methane and/or ethane. The product obtained by self-condensation of decaborane has properties which may permit its use as an aircraft fuel.

The following two equations were developed from which may be calculated the time required to decompose 20 percent of either decaborane or ethyl decaborane at any other temperature:

Decaborane:

$$\log (t_{20\%} - 2) = \frac{8.6 \times 10^3}{T} - 16.0$$

HEF-3:

$$\log (t_{20\%} - 2) = \frac{7.12 \times 10^3}{T} - 13.3$$

where T is temperature in °K and $t_{20\%}$ is time in minutes required for decomposition of 20 percent of the sample.

INTRODUCTION

A study of the thermal stabilities of decaborane and of ethyl decaborane (HEF-3) were undertaken at the NACA Lewis laboratory to supply data pertinent to fuel-system design and fuel storage. This study was made to obtain knowledge of the thermal stability of an alkylated decaborane and to compare this stability with the previously measured stability (ref. 1) of pentaborane. This work also measured the stability of decaborane for comparison with the alkylated decaborane and with pentaborane.

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Measurements were made of the amount of nonvolatile decomposition products formed when HEF-3 or decaborane was heated for various times at temperatures of 202° and 252° C.

APPARATUS AND PROCEDURE

The HEF-3 or decaborane was heated in a cylindrical stainless-steel bomb 3 inches long having an outside diameter of 5/8 inch and an inside diameter of 11/32 inch. The volume was approximately 1/4 cubic inch. The bomb was closed with a 1/8-inch stainless-steel Hoke valve and heated in an aluminum block furnace, which fitted closely about the bomb. The furnace was electrically heated and suitably insulated. A thermocouple well extended through the block to the midpoint on the bomb.

The HEF-3 or decaborane (1.3 g) was placed directly in the weighed bomb. This transfer was performed in an inert atmosphere. The bomb was sealed, evacuated, weighed, and placed in the furnace for the desired time. After removal from the furnace, the bomb was quenched and the volatile material was removed at a temperature of 110° C on a vacuum system until constant weight was obtained. The distillation temperature was below the temperature of decomposition. The bomb was reweighed to determine the nonvolatile residue. The percent decomposition was calculated by dividing the weight of nonvolatile residue by the weight of nonvolatile residue that was found after complete decomposition of a sample of fuel.

RESULTS AND DISCUSSION

Curves of the percent of decomposition plotted against time are shown in figure 1 for HEF-3, decaborane, and pentaborane at a temperature of 202° C. The data for pentaborane are taken from reference 1. The curves for HEF-3 and decaborane show a nearly steady rate of formation of nonvolatile residue with increase in time in contrast to the decrease in rate which is observed for pentaborane. Curves for the percent decomposition plotted against time at a temperature of 252° C for HEF-3 and decaborane are shown in figure 2. The experiments at 252° C were carried out for a time sufficient to decompose the sample completely. These curves also show a nearly steady rate of decomposition.

Figure 3 shows a plot of the reciprocal of Kelvin temperature against the $\log(t_{20\%} - 2)$ where $t_{20\%}$ is the time in minutes required for decomposition of 20 percent of the sample. Two minutes has been subtracted from the time required for the decomposition of 20 percent of the sample in order to remove from the calculation the approximate time required to bring the sample up to reaction temperature (ref. 1).

These data may be represented by the following equations:

Decaborane:

$$\log (t_{20\%} - 2) = \frac{8.6 \times 10^3}{T} - 16.0$$

HEF-3:

$$\log (t_{20\%} - 2) = \frac{7.12 \times 10^3}{T} - 13.3$$

Pentaborane:

$$\log (t_{20\%} - 2) = \frac{7.63 \times 10^3}{T} - 15.4$$

where T is temperature in $^{\circ}\text{K}$. The times required for other percentages of decomposition (i.e., 5 or 10 percent) may be estimated by multiplying the time required for 20-percent decomposition by

$$\frac{\text{percent decomposition}}{20 \text{ percent decomposition}}$$

This calculation is based on the assumption of a straight-line dependence between the percent decomposition and time; this assumption will not lead to a large absolute error if used between about 1 or 2 percent and 75 percent. In addition, since the experimental points lie below the straight lines of figures 1 and 2, the use of this relation will give a percent of decomposition which is somewhat more than will actually be obtained.

The physical properties of the residue which is obtained from HEF-3 or decaborane are different from those of the residue from pentaborane. The solid product from the pyrolysis of pentaborane (ref. 1) or ethyl pentaborane (ref. 2) has no softening or melting point below the temperature of its own decomposition into boron and hydrogen. Also, the residue obtained from pentaborane, either in its light yellow colored modification formed at lower temperatures or in its darker yellow modification formed at higher temperatures, has no appreciable solubility in pentaborane. In contrast, the material formed from the decomposition of decaborane is a viscous liquid which is soluble in alkylated decaborane or in liquid decaborane. The product formed from the decomposition of HEF-3 is generally similar in solubility to the product obtained from decaborane. The solubility of these products in alkylated decaborane will decrease fuel-line clogging below that observed with pentaborane. Keeping the fuel hot but below its temperature of rapid decomposition will further decrease the precipitation of residues. The increased temperature will also decrease the viscosity of either the residue or the solution of the residue in fuel.

Similar to the experience with pentaborane, pressure buildup occurs with decaborane and HEF-3 because of the liberation of hydrogen from decaborane and of hydrogen and ethane and/or methane from ethyl decaborane. Analysis of the residue reveals that nearly all the carbon is lost to the gas phase as ethane and/or methane. Hydrogen is lost until the residue has a boron-hydrogen ratio of approximately 1. From these results, the maximum pressure buildup may be calculated for any amount of decomposition of ethyl decaborane and for a known tank ullage. Below 202° C pressure buildup is slow and at normal temperatures of storage is sufficiently slow to permit storage to be a practical operation.

This study of the decomposition of decaborane was part of the investigation of the self-condensation of decaborane. The investigation had as its objective the discovery of new boron hydrides which might have properties suitable for aircraft propulsion. The product which is formed by this condensation with elimination of hydrogen does not hydrolyze appreciably in water. The product is soluble in ethyl decaborane. Consistency of the product varies from a viscous liquid to a sticky solid. The product may be used as an aircraft fuel or blended with ethyl decaborane to increase the heating value of the ethyl decaborane.

CONCLUSIONS

The results indicate that a commercial grade of ethyl decaborane (HEF-3) is more stable to pyrolysis than pentaborane but less stable than decaborane.

Evaluation of the physical properties of the product obtained from the self condensation of decaborane indicate that this material may be used as a fuel for aircraft propulsion.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
Cleveland, Ohio, September 24, 1956

REFERENCES

1. McDonald, Glen E.: The Rate of Decomposition of Liquid Pentaborane from 85° to 202° C. NACA RM E55H01, 1955.
2. McDonald, Glen E.: Thermal Decomposition of Ethylpentaborane in Gas Phase. NACA RM E56D26, 1956.

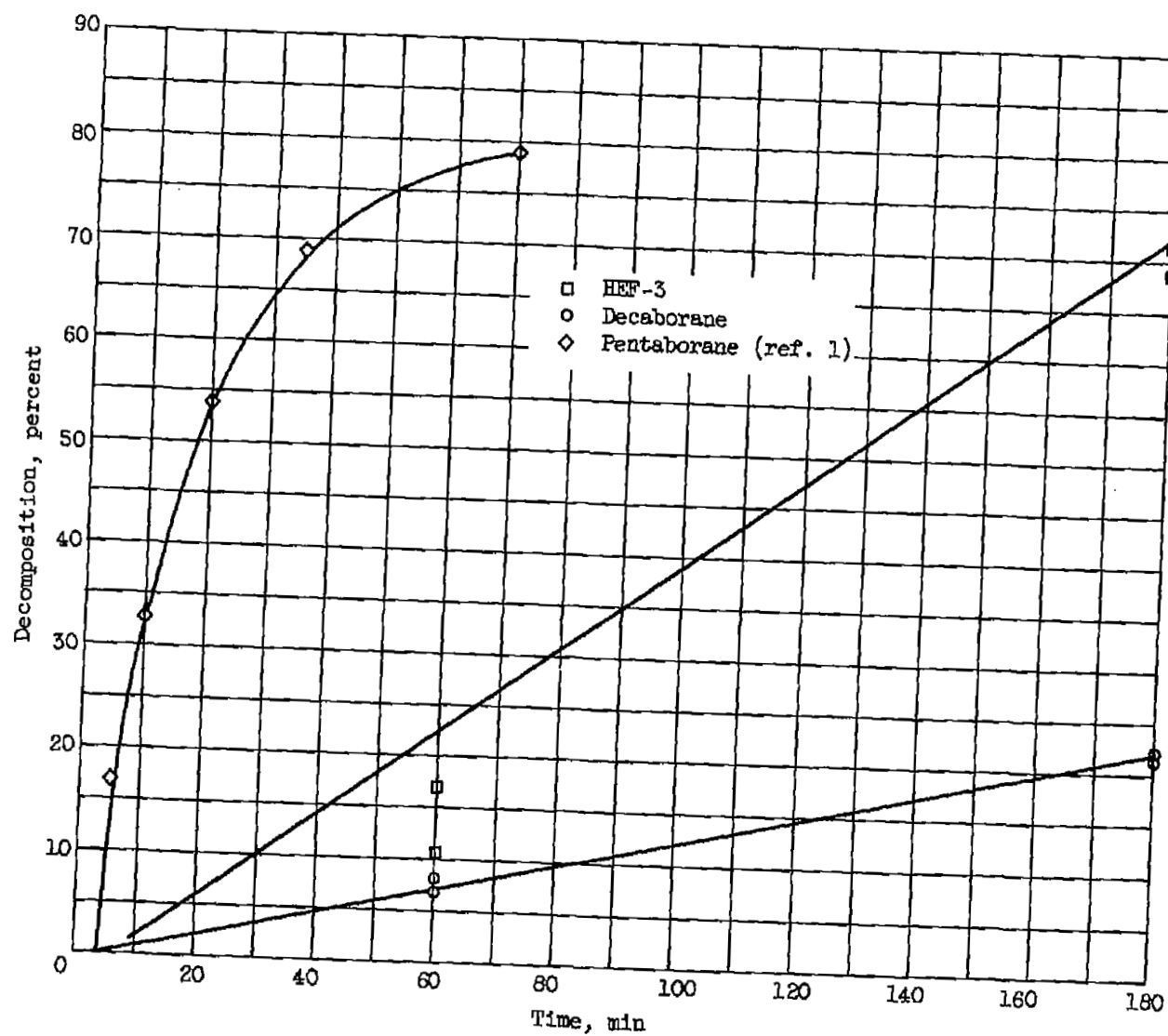


Figure 1. - Decomposition of HEF-3, pentaborane, and decaborane at 202° C.

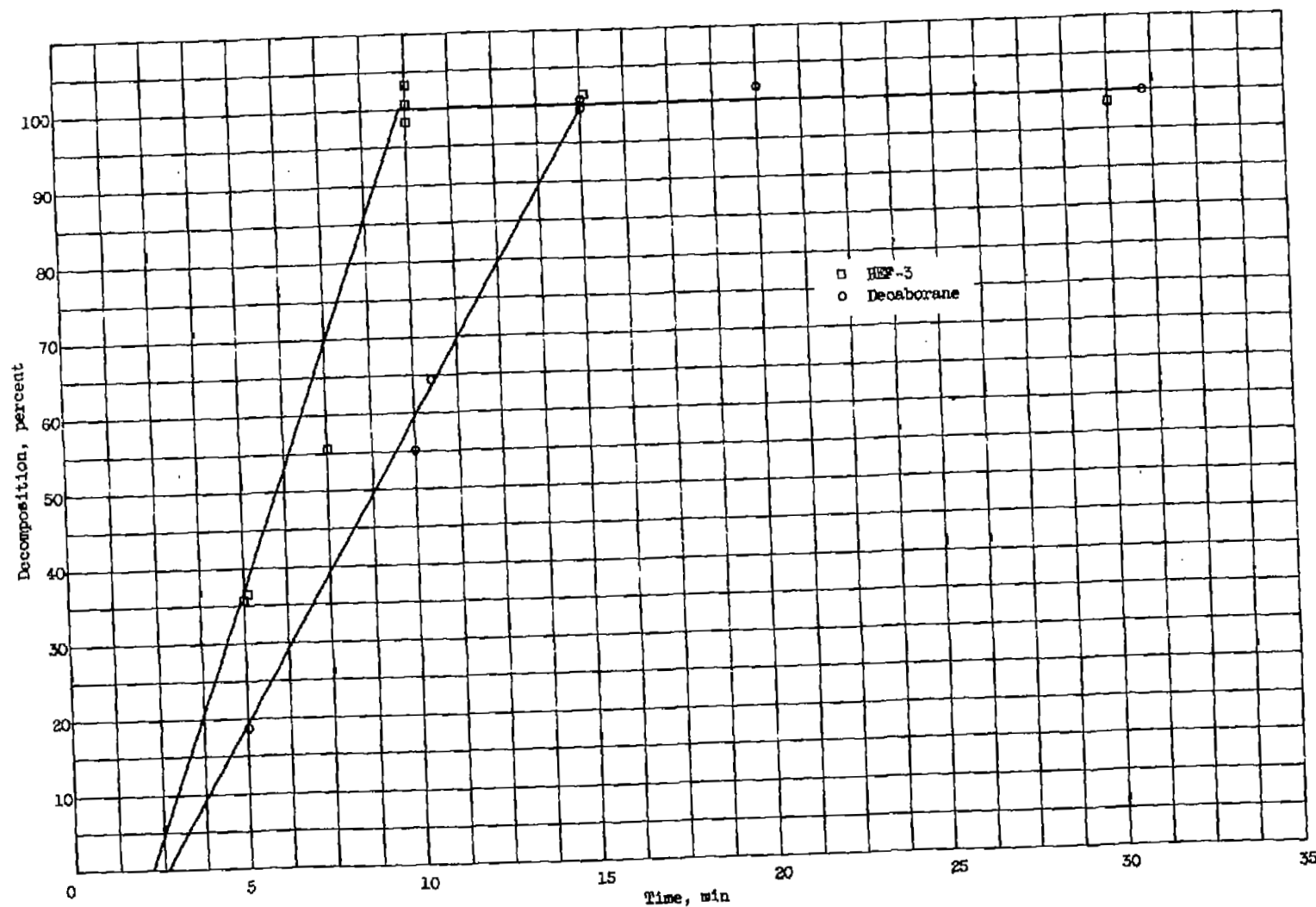


Figure 2. - Decomposition of HEP-3 and decaborane at 252° C.

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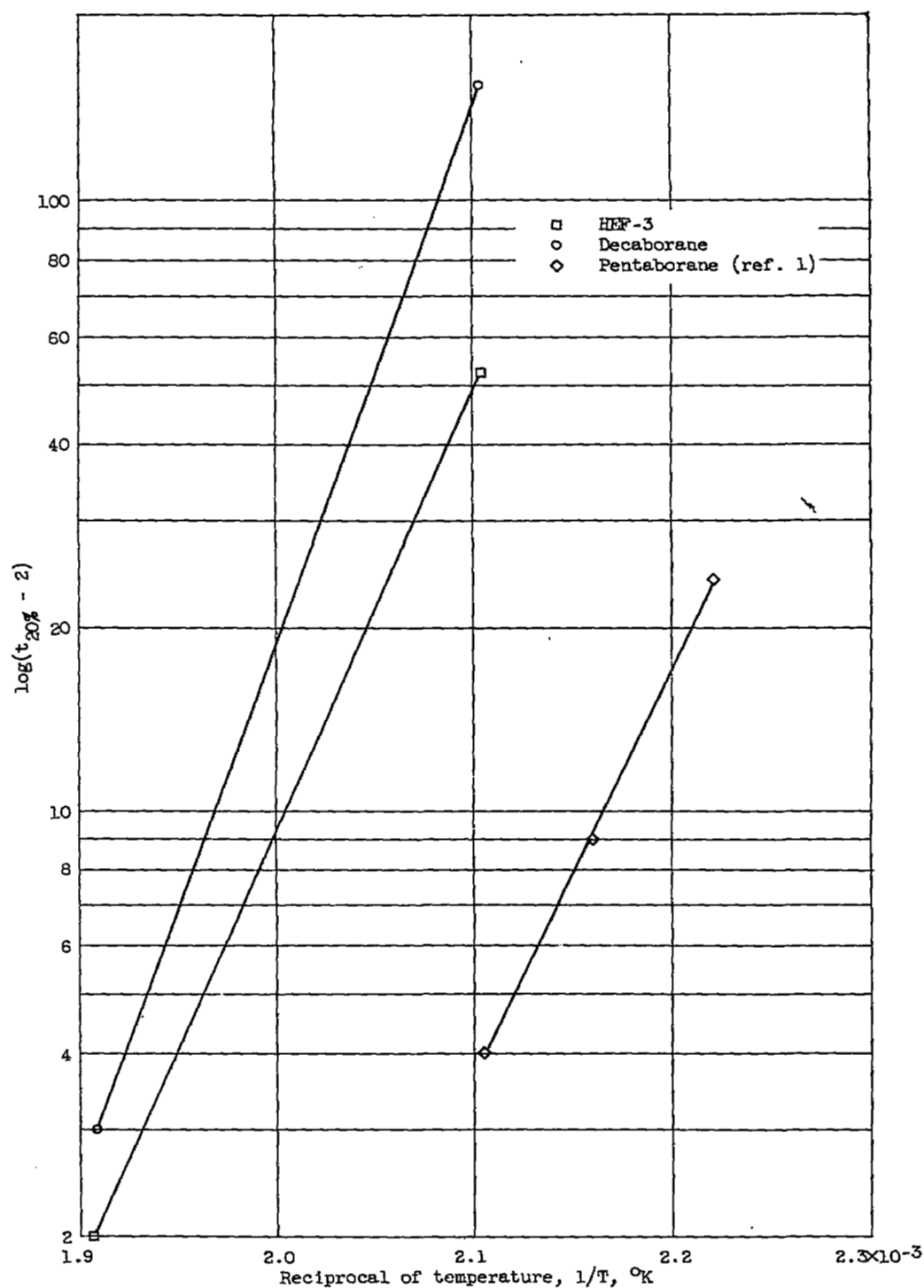


Figure 3. - Reciprocal temperature plotted against logarithm of time required for 20 percent decomposition.

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